of redistilled **2** (0.137 mol) and 50 ml of freshly redistilled diglyme was stirred and refluxed for 45 min under N_2. The cooled reaction mixture was steam distilled and the 300 ml of pale yellow distillate obtained was saturated with NaCl and extracted three times with ether. The ether solution was dried ($MgSO_4$), concentrated and distilled to yield 7.56 g of oil, bp 52–70° (19 mm). Glpc analysis on a 5 ft × 0.25 in. column packed with 30/60 mesh Chromosorb W containing 15% Carbowax 20M indicated three compounds: isobutyraldehyde,[9] 0.3%, retention time (R_T) at 107°, 1.0 min; diglyme, 88%, R_T 15.2 min; and **8**, 11%, R_T 10.4 min. Samples of each substance were condensed from the glpc effluent (He) at Dry Ice temperature for characterization. Isobutyraldehyde and diglyme were identified by R_T and by their ir spectra. Compound **8** was a colorless, mobile liquid with a peculiar vegetablelike odor: ir 3.40, 3.50, 6.0 (C═N), 6.85, 7.00, 7.25, 7.30, 7.85, 8.15, 9.10, 9.30, 10.00 and 10.50 μ; 100 MHz nmr (CCl_4) δ 0.94 [d, 6, J = 6 Hz, $(\mathbf{CH_3})_2CH$], 1.29 (d, 3, J = 6 Hz, $CH\mathbf{CH_3}$), 1.78 (broad m, 1, J = 6 Hz, >**CH**–), 1.92 and 1.94[10] (s, 3, $\mathbf{CH_3}C$═), 4.48 (broad q, 1, J = 6 Hz, >$\mathbf{CH}CH_3$) and 5.08 ppm [m, 1, –(O)**CH**N═]; mass spectrum (70 eV) m/e 141 (molecular ion) 139, 124, 98 (base peak), 97, 82, 71, 55, 56, 43, 42, 41, 39.

Anal. Calcd for $C_8H_{15}NO$: N, 9.92. Found: N, 9.9.

Treatment of the original distillation residue with excess picric acid in ethanol at 25° gave 3.62 g (9%) of **9** dipicrate, mp 185–190°. Recrystallization from ethanol gave yellow needles, mp 196.5–198° (lit.[11] mp 198.5–199.5°).

Anal. Calcd for $C_{20}H_{18}N_8O_{14}$: C, 40.41; H, 3.05; N, 18.84. Found: C, 39.9; H, 3.0; N, 18.7.

Decomposition of the picrate with aqueous NH_3 gave pure **9** whose ir spectrum (CS_2) was identical with that of an authentic specimen.

Chloranil Dehydrogenation of 8.—A solution of **8** (0.380 g, 0.00270 mol) in 4 ml of diglyme was treated with 0.749 g (0.00304 mol) of freshly recrystallized chloranil. The mixture was stirred and heated to 100° for 1 hr. On cooling, 0.688 g of solid (presumably tetrachlorohydroquinone) was filtered off and the filtrate was distilled. A single product was formed which codistilled with diglyme, bp 60.5–64.5° (14 mm). The compound was separated by preparative glpc using the column described above and was shown to be 2-isopropyl-4,5-dimethyloxazole by comparing ir and R_T data with those of the authentic substance. The oxazole was formed in 80% yield.

2-Isopropyl-4,5-dimethyloxazole was prepared by the method of Theilig[12] from isobutyramide and 3-bromo-2-butanone (Eastman Organic Chemicals, Rochester, N.Y.). The compound was a colorless liquid: bp 76–77° (20 mm); yield 70%; ir (CH_2Cl_2) 3.45, 6.05, 6.40, 7.25, 8.35, 8.85, 9.15, 9.40, 10.15 and 10.50 μ; 100 MHz nmr ($CDCl_3$) δ 1.28 [d, 6, J = 7 Hz, $(\mathbf{CH_3})_2CH$], 2.00 (s, 3, ring CH_3), 2.14 (s, 3, ring CH_3) and 2.94 ppm [m, 1, J = 7 Hz, $(CH_3)_2\mathbf{CH}$].

Anal. Calcd for $C_8H_{13}NO$: C, 69.03; H, 9.41; N, 10.06. Found: C, 68.6; H, 9.2; N, 9.5.

N-(1-Methyl-2-oxopropylidene)isobutylamine (**10**).—A solution containing 4.00 ml (0.0457 mol) of **2** in 100 ml of benezene was treated with 4.55 ml (0.0457 mol) of isobutylamine and refluxed under a Dean-Stark water separator for 1 hr (0.92 ml of water separated). The benezene solution was concentrated and the residue was distilled giving 3.13 g (49%) of **10**: bp 65–67° (16 mm); ir 3.40, 5.85 (C═O), 6.10 (C═N), 6.80, 7.40, 7.75, 9.00 and 10.20 μ; 60 MHz nmr ($CDCl_3$) δ 0.95 [d, 6, J = 6 Hz, $(\mathbf{CH_3})_2CH$], 1.80 and 1.82 [s, 4, (includes isopropyl methine H), $\mathbf{CH_3}C$═N], 1.93 (m, J = 6 Hz, >$\mathbf{CH}CH_2$), 2.22 (s, 3, $\mathbf{CH_3}C$(═O)–), 3.10 and 3.12[13] (d, 2, J = 7 Hz, ═N$\mathbf{CH_2}$).

Anal. Calcd for $C_8H_{15}NO$: C, 68.04; H, 10.71; N, 9.92. Found: C, 68.4; H, 10.6; N, 10.3.

Registry No.—**1**, 516-06-3; **2**, 431-03-8; **8** (*cis*), 19519-42-7; **8** (*trans*), 19519-43-8; **9**, 1124-11-4; **10**, 19519-44-9; 2-isopropyl-4,5-dimethyloxazole, 19519-45-0.

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[9] No attempt was made to recover the bulk of the isobutyraldehyde which was presumably lost during distillation.

[10] Two nearly superimposed sharp singlets were observed, apparently due to a difference in chemical shifts of the 4-methyl group hydrogens in *cis* and *trans* **8**. The isomers of **8** were partially resolved on a 10 ft × 0.125 in. glpc column packed with 60/80 mesh Chromosorb W (HMDS treated) containing 15% SF-96.

[11] T. Ishiguro, E. Kitamura, and M. Matsumura, *Yakugaku Zasshi*, **78,** 150 (1959); *Chem. Abstr.*, **53,** 13163 (1959).

[12] G. Theilig, *Chem. Ber.*, **86,** 96 (1953).

[13] Two singlets and two doublets believed due to *syn* and *anti* forms of the imine; *cf.* G. J. Karabatsos and S. S. Lande, *Tetrahedron*, **24,** 3907 (1968).

Addition of Thiobenzophenone to Benzenediazonium-2-carboxylate[1]

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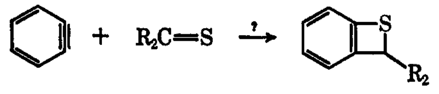
One possible way to prepare benzothietes[2] is by a 1,2 cycloaddition of a thiocarbonyl group to benzyne (1,2-dehydrobenzene). Examples are known of the cycloaddition of a thiocarbonyl group to double bonds,[3] but the only reported interaction with a benzyne occurs with formation of a benzothiazole.[4] A number of 1,2 cycloadditions of benzyne with other types of compounds are known.[5]



To check the feasibility of adding thiocarbonyl groups to benzyne, thiobenzophenone and propylene oxide were added to a solution of the hydrochloride of benzenediazonium-2-carboxylate in 1,2-dichloroethane[6] and the solution was refluxed. Gas and heat were evolved and a white solid was obtained (44.5% yield, purified) which was identified as 2,2-diphenyl-3,1-benzoxathian-4-one (**1**), the δ-lactone of *o*-[(α-hydroxybenzhydryl)thio]benzoic acid, which has not been prepared before although a number of 3,1-benzoxathian-4-ones have been synthesized by other methods.[7] The identification was accomplished by determination of the compound's molecular weight, its empirical formula by analysis for elements, its mass spectrum, infrared

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[1] This work was aided by Grant GP 5513 of the National Science Foundation and by Grant CA 08250 of the National Cancer Institute, National Institutes of Health.

[2] These compounds are interesting because of the possibility that their anions might show relative stabilization (they are formally 10-π-electron systems). The intervention of an anion in the reduction of a naphthothiete sulfone has been considered: D. C. Dittmer and N. Takashina, *Tetrahedron Lett.*, 3809 (1964). Several substituted benzothiete derivatives not suited for the preparation of thiete anions have been prepared by the reduction of sulfones: L. A. Paquette, *J. Org. Chem.*, **30,** 629 (1965).

[3] H. Staudinger, *Helv. Chim. Acta*, **3,** 862 (1920); E. T. Kaiser and T. F. Wulfers, *J. Amer. Chem. Soc.*, **86,** 1897 (1964); W. J. Middleton, *J. Org. Chem.*, **30,** 1395 (1965); G. Tsuchihashi, M. Yamauchi, and M. Fukuyama, *Tetrahedron Lett.*, 1971 (1967); P. Rioult and J. Vialle, *Bull. Soc. Chim. Fr.*, 2883 (1967); K. Yamada, M. Yoshioka and N. Sugiyama, *J. Org. Chem.*, **33,** 1240 (1968).

[4] B. F. Hrutford and J. F. Bunnett, *J. Amer. Chem. Soc.*, **80,** 2021 (1958).

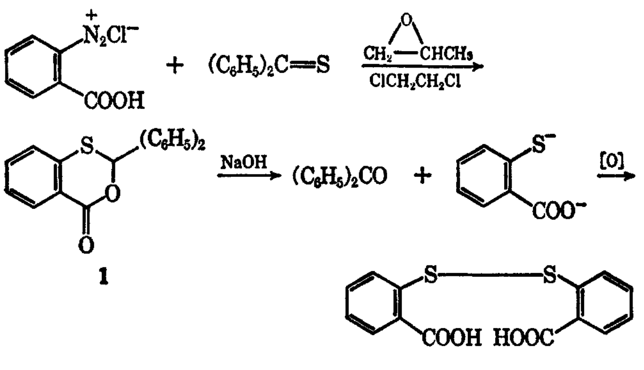
[5] (a) Reviewed by R. W. Hoffmann, "Dehydrobenzene and Cycloalkynes," Academic Press, New York, N. Y., 1967; (b) L. L. Muller and J. Hamer, "1,2-Cycloaddition Reactions," Interscience Publishers, New York, N. Y., 1967.

[6] For this method of preparation of benzyne, see L. Friedman and F. M. Logullo, *J. Amer. Chem. Soc.*, **85,** 1549 (1963).

[7] (a) D. T. Mowry, W. H. Yanko and E. L. Ringwald, *ibid.*, **69,** 2358 (1947); (b) A. Senning and S.-O. Lawesson, *Acta Chem. Scand.*, **14,** 2230 (1960); *Arkiv Kemi*, **17,** 261, 387, 489 (1961), and **18,** 95 (1961); (c) W. G. Bentrude and J. C. Martin, *J. Amer. Chem. Soc.*, **84,** 1564 (1962). See also references cited in these publications.

spectrum, ultraviolet spectrum, and proton nmr spectrum. Hydrolysis with base gave benzophenone, which was identified as its 2,4-dinitrophenylhydrazone, and 2,2′-dicarboxyphenyl disulfide, derived from 2-mercaptobenzoic acid which is oxidized by air (Scheme I). The disulfide was identical with a known sample with respect to its melting point and infrared (ir) spectrum.

SCHEME I

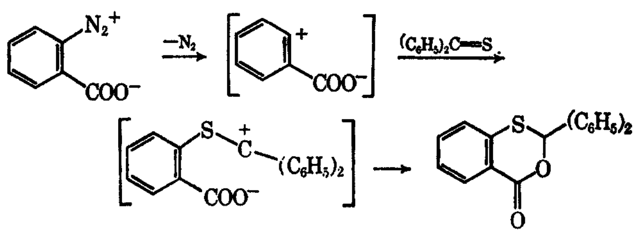


The mass spectrum of the benzoxathian-4-one had a prominent parent peak (P) at *m/e* 318. The base peak was at *m/e* 136 [P − $(C_6H_5)_2CO$], and other prominent peaks were at *m/e* 108 [P − $(C_6H_5)_2COCO$] and *m/e* 105 [P − $(C_6H_5)_2COS$ + H)] These fragments support the structure assignment.

Absorption in the ir spectrum at 1730 cm^{-1} is consistent with the absorption at 1724 cm^{-1} (5.80 μ) reported for several 3,1-benzoxathian-4-ones.[7b] The ultraviolet (uv) spectrum in methanol showed absorptions at 233, 265, and 330 mμ which are not inconsistent with those expected of an *o*-thio-substituted benzoic acid derivative.[8] The proton nmr spectrum showed only absorption centered at δ 7.4, attributable to aromatic protons.

Formation of 2,2-diphenyl-3,1-benzoxathian-4-one suggests that an intermediate in benzyne formation from benzenediazonium-2-carboxylate is being trapped by the thiobenzophenone. A possible mechanism is shown in Scheme II. Recently, evidence has been obtained for a two-step mechanism in the formation of benzyne[9] and there have been earlier indications of the nonsynchronous loss of nitrogen and carbon dioxide from benzenediazonium-2-carboxylate.[10]

SCHEME II



Experimental Section

Benzenediazonium-2-carboxylate Hydrochloride.[6]—Anthranilic acid (10.95 g, 8 × 10^{-2} mol) was dissolved in 120 ml of absolute ethanol in a 400-ml beaker. The solution was stirred magnetically in a dish containing ice. To this was added 8 ml of concentrated hydrochloric acid followed by 20 ml of isoamyl nitrite. The reaction mixture was stirred for 10 min and 120 ml of absolute ether was added. Pale pink crystals precipitated from solution as stirring was continued for an additional 5 min. The crystals were filtered through a polyethylene funnel and washed with 150 ml of absolute ether. The product was dried in air to give 14.1 g (7.58 × 10^{-2} mol, 95%).

2,2-Diphenyl-3,1-benzoxathian-4-one.—A 250-ml, three-necked, round-bottomed flask was fitted with a condenser, ground-glass stopper, and a standard taper stopcock. The flask was swept with nitrogen for 20 min and the flow was continued throughout the reaction. A solution of benzenediazonium-2-carboxylate hydrochloride (4.0 g, 2.17 × 10^{-2} mol) in 100 ml of 1,2-dichloroethane was added to the flask. In rapid succession thiobenzophenone[11] (5.0 g, 2.52 × 10^{-2} mol) and propylene oxide (3.9 ml, 5.46 × 10^{-2} mol) were added, and the reaction mixture was refluxed for 24 hr. The solution turned wine-red 0.5 hr after the final addition was complete. The reaction mixture was cooled and the solvent was removed by means of a rotating evaporator leaving a wine-red oil. The oil was dissolved in a minimum amount of benzene and chromatographed on a Florisil (100–200 mesh) column. Elution with benzene gave a number of bands. A blue and purple band ran together and came off the column first. The solvent was evaporated over a steam bath in a nitrogen atmosphere leaving a deep violet oil. This oil was dissolved in a minimum amount of benzene and chromatographed on a Florisil (100–200 mesh) column. Elution with 2:1 ligroin–benzene afforded the following bands in their order of elution: dark blue, tan, clear, orange, and pink.

The first band yielded a blue oil after the evaporation of solvent under nitrogen. Recrystallization from petroleum ether afforded 1.12 g of deep violet needles, mp 51–52° (uncor), identified as thiobenzophenone by mixture melting point, 51–52° (lit.[11] mp 53–54°), with an authentic sample. This represents a return of 5.7 × 10^{-3} mol or 22.4% of the starting material.

After the evaporation of solvent the tan band left a pale tan oil. Crystallization and subsequent recrystallization from ethanol yielded 0.17 g (1.13 × 10^{-3} mol, 5.2%) of tan solid, mp 109° (uncor). The solid was identified as biphenylene by comparison of infrared (ir) spectra and mixture melting point,[12] 108.5–109° (lit.[13] mp 109–110°).

The clear band afforded a white solid after the removal of solvent. Two recrystallizations from methanol yielded 3.16 g (9.95 × 10^{-3} mol, 44.5%) of pure 2,2-diphenyl-3,1-benzoxathian-4-one: mp 185° (uncor); λ_{max}^{MeOH} 233, 265, and 330 mμ.

Anal. Calcd for $C_{20}H_{14}O_2S$: C, 75.47; H, 4.40; O, 10.10; S, 10.10; mol wt, 318. Found: C, 75.67; H, 4.40; O, 10.00; S, 9.89; mol wt, 320 (osmometric).

Molecular weight determination using the mass spectrometer gave a value of 318. The ir absorptions are as follows: 1730 (s), 1600 (m), 1490 (m), 1480 (m), 1445 (m), 1270 (m), 1240 (m), 1215 (m), 1170 (m), 1125 (m), 1105 (m), 1050 (m), 1030 (m), 1000 (m), 960 (w), 955 (w), 938 (w), 928 (w), 915 (w), 905 (w), 887 (w), 857 (w), 850 (w), 835 (w), 819 (w), 810 (w), 797 (m), 753 (s), 747 (s), 720 (w), and 695 (s) cm^{-1}. The nmr spectrum shows only one absorption, a multiplet centered at δ 7.4 (60 MHz).

Hydrolysis of 2,2-Diphenyl-3,1-benzoxathian-4-one.—A 50-ml, one-necked, round-bottomed flask was fitted with a condenser and magnetic stir bar. A mixture of 15 ml of 1,4-dioxane and 15 ml of 6 *N* sodium hydroxide was placed in the flask. To this was added 2,2-diphenyl-3,1-benzoxathian-4-one (0.376 g, 1.18 × 10^{-3} mol) and the solution was refluxed for 15 hr. The reaction mixture was cooled and the aqueous layer was separated. The organic layer was washed twice with two 20-ml portions of 6 *N* sodium hydroxide which were added to the aqueous layer collected previously. This was acidified with concentrated hydro-

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(8) The uv spectrum of *o*-thioalkyl benzoate esters appear not to have been reported, but (2-carboxyphenyl)thioglycolic acid is reported to have maxima at 223, 258, and 316 mμ and *o*-mercaptobenzoic acid has absorption at 220 and 313 mμ: "Absorption Spectra in the Ultraviolet and Visible Region," Vol. 1, L. Lang, Ed., Academic Press, New York, N. Y., 1961, pp 95, 99.

(9) R. Gompper, G. Seybold, and B. Schmolke, *Angew. Chem. Intern. Ed. Engl.*, **7**, 389 (1968).

(10) M. Stiles and R. G. Miller, *J. Amer. Chem. Soc.*, **82**, 3802 (1960); S. Yaroslavsky, *Tetrahedron Lett.*, 1503 (1965); R. Knorr, *Chem. Ber.*, **98**, 4038 (1965); S. Yaroslavsky, *Chem. Ind.* (London), 765 (1965); M. Kise, T. Asari, N. Furukawa and S. Oae, *ibid.*, 276 (1967). See also ref 5a.

(11) B. F. Gofton and E. A. Braude, "Organic Syntheses," Coll. Vol. IV, John Wiley & Sons, Inc., New York, N. Y., 1963, p 927.

(12) We are indebted to J. M. Balquist for an authentic sample and spectrum of biphenylene.

(13) W. C. Lothrop, *J. Amer. Chem. Soc.*, **63**, 1187 (1941).

chloric acid. A white inorganic solid, which did not burn, precipitated on cooling. Its ir spectrum showed the following absorptions: 3000 (m), 2640 (m), 2540 (m), 1675 (s), 1580 (m), 1550 (m), 1455 (m), 1425 (m, sh), 1410 (m), 1305 (m), 1280 (m), 1255 (s), 1200–1030 (s, broad), 900 (m), 810 (m), 740 (s), and 690 (m) cm^{-1}. The solid was refluxed for 24 hr in 50 ml of concentrated hydrochloric acid. A tan solid precipitated when the solution was cooled. The filtrate was washed with two 50-ml portions of chloroform which were combined and evaporated to dryness leaving an additional amount of the tan solid. Recrystallization from methanol-water afforded 0.287 g (8.98×10^{-4} mol, 79.9%) of pure tan solid, mp 302–303° (uncor). The ir spectrum showed the following absorptions: 3000–2800 (m), 2640 (w), 2540 (w), 1680 (s), 1580 (m), 1555 (m), 1455 (m), 1410 (m), 1305 (m), 1285 (m), 1255 (s), 1165 (w), 1145 (m), 1115 (w), 1055 (m), 1035 (m), 960 (w), 900 (m, broad), 810 (m), 740 (s), and 693 (m) cm^{-1}. Based on the ir spectrum and a mixture melting point, 301.5–302.5° (authentic sample from Aldrich Chemical Co., mp 304°), the compound was identified as 2,2′-dicarboxyphenyl disulfide.

The dioxane was removed by means of a rotating evaporator leaving 0.153 g (8.42×10^{-4} mol, 71.2%) of a clear oil. A comparison of the ir spectrum of the oil with an authentic sample of benzophenone indicated that the two compounds were identical. Treatment with an acidic solution of 2,4-dinitrophenylhydrazine gave a bright orange solid. This was dissolved in a minimum amount of chloroform and chromatographed on a Florisil (100–200 mesh) column. The column was eluted with chloroform. Two bands separated with the first being bright orange and the second a deep red. After evaporation of solvent and recrystallization from chloroform–ethanol the first band afforded benzophenone 2,4-dinitrophenylhydrazone, mp 237–238°, mmp 237–238°. An authentic sample of the 2,4-dinitrophenylhydrazone was prepared from benzophenone and its ir spectrum and melting point (238°) were identical with those of the hydrazone obtained from the product of hydrolysis of the 2,2-diphenyl-3,1-benzoxathian-4-one. The red band afforded 2,4-dinitrophenylhydrazine, mp 199°.

Registry No.—Thiobenzophenone, 1450-31-3; benzenediazonium-2-carboxylate, 18761-40-5; 1, 19185-81-0.

2,4,5-Triphenyl-2H-1,3-oxathiole from Desyl Thiocyanate[1]

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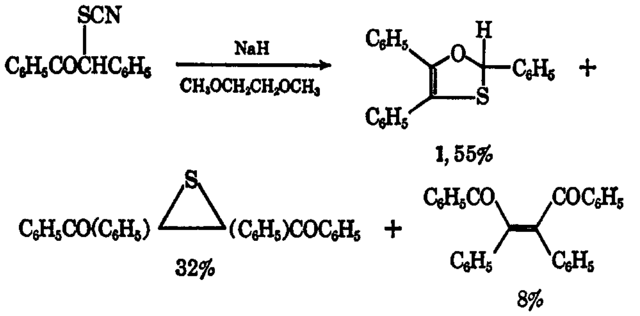
A pale yellow solid, $C_{21}H_{16}OS$ (55% yield, mp 77–78°), was obtained when desyl thiocyanate (2-thiocyanato-2-phenylacetophenone) was treated with excess sodium hydride in 1,2-dimethoxyethane. *cis*-Dibenzoylstilbene episulfide (2,3-dibenzoyl-2,3-diphenylthiirane, 32%) and *cis*-dibenzoylstilbene (8%) were isolated also from the reaction mixture. When the sodium hydride was not in excess, a 75–85% yield of yellow *cis*-dibenzoylstilbene episulfide was obtained, as had been reported previously.[2]

The data for compound $C_{21}H_{16}OS$ are consistent with the structure 2,4,5-triphenyl-2H-1,3-oxathiole (**1**). Few simple 1,3-oxathioles are known,[3] and this new method may be applicable generally to the synthesis of 2,4,5-triaryl-1,3-oxathioles. The structure of the oxathiole was deduced from an analysis for elements, a molecular weight determination, the infrared, ultraviolet, and proton nmr spectra, and the mass spectrum. The infrared spectrum showed absorption at 3030 (aromatic protons), 2880 (benzylic proton), 1620 (carbon–carbon double bond), 1245 (asymmetric carbon–oxygen stretching), and 1060 cm^{-1} (symmetric carbon–oxygen stretching). Absorption in the infrared spectrum of 2-trichloromethyl-4,5-diphenyl-1,3-dioxole at 1667, 1250, and 1124 cm^{-1} has been ascribed to the carbon–carbon double bond and to the carbon–oxygen bonds, respectively.[4] In 1,3-dioxole itself there is absorption at 1631, 1176–1156, and 1087–1075 cm^{-1}.[5] The ultraviolet spectrum of the oxathiole in 95% ethanol shows maxima at 225 mμ (ϵ 18,600) and 342 mμ (ϵ 6760). The ultraviolet spectrum of 5,6-dihydro-1,4-oxathiin has an absorption maximum at 229 mμ (ϵ 3820)[6] and 2,3-diphenyl-5,6-dihydrooxathiin is reported to be pale yellow[7] but spectroscopic data were not given.



The proton nmr spectrum (60 MHz, CDCl$_3$) shows a complex multiplet at 433 Hz and a singlet at 418 Hz relative to tetramethylsilane. The ratio of the areas of the two absorptions was 14.4:1 which is close to the 15:1 ratio calculated for the oxathiole. The benzylic proton presumably causes the absorption at 418 Hz; it is at low field because it is adjacent to an oxygen atom, a sulfur atom, and a phenyl ring. Diamagnetic anisotropy effects of the carbon–carbon double bond, the sulfur atom, and the phenyl rings probably have a role in deshielding the benzylic proton. The absorption of the aliphatic proton in 2-trichloromethyl-4,5-diphenyl-1,3-dioxole appears at 373 Hz.[4]

Mass spectrometry (Scheme I) supports the molecular formula $C_{21}H_{16}OS$. The molecular ion (P) was also the base peak and prominent fragments occurred at *m/e* 284 (P − S), 283 (P − SH), 239 (P − C_6H_5), 211 (P − C_6H_5CO), 210 (P − C_6H_5CHO), 178 (P − C_6H_5CHO − S), 167 and 165. The fragments at *m/e* 165 and 167 may be fluorenyl-type ions whose formation would involve a migration of a phenyl group. Treatment of a small amount of the oxathiole with refluxing ethanolic hydrogen chloride gave hydrogen

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[1] This work was aided by Grant GP-5513 of the National Science Foundation and by Grant CA 08250 of the National Cancer Institute, National Institutes of Health.

[2] D. C. Dittmer and G. C. Levy, *J. Org. Chem.*, **30**, 636 (1965); D. C. Dittmer, G. E. Kuhlmann, G. C. Levy, and R. Keene, paper in preparation.

[3] For a recent review of 1,3-oxathioles, see D. S. Breslow and H. Skolnik, "Heterocyclic Compounds, Multisulfur and Sulfur and Oxygen Five- and Six-Membered Heterocycles," Part One, A. Weissburger, Ed., Interscience Publishers, Inc., New York, N. Y., 1966, p 203 ff.

[4] H. J. Dietrich and J. V. Karabinos, *J. Org. Chem.*, **31**, 1127 (1966).

[5] N. D. Field, *J. Amer. Chem. Soc.*, **83**, 3504 (1961).

[6] W. E. Parham, I. Gordon, and J. D. Swalen, *ibid.*, **74**, 1824 (1952).

[7] J. R. Marshall and H. A. Stevenson, *J. Chem. Soc.*, 2360 (1959).